AN ATTEMPT TO ISOLATE THE PRECURSORS OF OFF FLAVOR IN OXIDIZED DEHYDRATED SWEET POTATO FLAKES

by

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Recent studies of the development of "off flavor" in dehydrated sweet potato flakes have implicated a lipid fraction containing a small amount of carotenoid epoxide. The fraction also appeared to contain other compounds which could be the precursors of such "off flavor." The major objective of this research was to isolate the compounds present in this lipid fraction and to determine the relationship of these compounds to "off flavor" in oxidized dehydrated sweet potato flakes.

A large fraction was carved out of a chromatographic column and subjected to further column chromatography and preparative thin layer chromatography before the samples were allowed to oxidize. All samples streaked on chromatographic columns and plates, indicating no pure compounds had been obtained. In an effort to simplify the mixture and obtain cleaner chromatographic separation, the total lipid extract was partitioned by use of a counter current apparatus and subjected to various methods of chromatography before oxidation. Sensory evaluation of the oxidized samples in comparison to good and "off flavored" reconstituted sweet potato flakes indicated little correlation between the odors of the samples and the odor of the reconstituted flakes.

A steam distillate prepared from reconstituted sweet potato flakes oxidized to produce an odor similar to "off flavored" sweet potato flakes. This distillate contained moisture, the removal of

which resulted in the loss of many volatile substances. Again no pure compounds were obtained as shown by the streaking of this distillate on chromatographic plates. Oxidized samples of this distillate when presented to a sensory panel were judged not to contain odors similar to reconstituted sweet potato flakes.

A short path molecular still was used to prepare distillate samples at temperatures lower than those used by steam distillation and to avoid the problem of subsequent separation of the volatiles from water. All distillate samples were subjected to chromatographic analysis before oxidation and presentation to a panel for sensory evaluation. Samples were prepared from raw and cooked Centennial variety sweet potatoes. No pure compounds were obtained by any of the analytical methods employed. Gas chromatographic and mass spectral analysis indicated that the distillate sample consisted of a complex mixture of previously unidentified compounds which were thought to be mainly complex terpenes.

Statistical analyses were performed on sensory panel data which indicated some similarity between the oxidized samples and the odor of the reconstituted "off flavored" flakes. Least significant difference values were calculated to give an indication of the difference required between two treatment means to show significance.

Sensory evaluations of purified fractions showed little correlation between sample odors and the odor of reconstituted "off flavored" sweet potato flakes until good reconstituted sweet potato flakes were added to the purified fractions. Sensory evaluations

then revealed some definite similarities of odor between the combined samples and the "off flavored" sweet potato flakes. Panel members also indicated that the "off flavored" samples were not highly objectionable to them.

The results obtained throughout this study led to the conclusions that:

1. The lipid and volatile fractions from sweet potatoes are highly complex compounds which cannot be separated into pure substances with analytical methods in current use.

2. Purification of compounds implicated in "off flavor" development resulted in a loss of any resemblance to "off flavored" sweet potato flakes.

3. Development of "off flavor" in sweet potato flakes is a highly complex reaction involving combinations of as yet unidentified compounds.

4. Many of the same odors present in "off flavored" sweet potato flakes are also present in good sweet potato flakes.

5. Oxidation of sweet potato fractions may reach a point beyond which all resemblance to "off flavored" sweet potato flakes is lost.

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CHAPTER I

INTRODUCTION

The ultimate purpose of food research is to make wholesome and nutritious food economically available at all seasons wherever required. Foods are complex organic mixtures further complicated by biological structures. Due to this complexity highly sophisticated research methods of several disciplines are required to determine basic chemical and physical attributes of foods. These attributes are essentially without meaning unless they can be related to wholesomeness and nutrition.

Sweet potato flakes developed almost simultaneously in the Food Science Department at North Carolina State University, Raleigh, North Carolina and at the Southern Utilization Research and Development Division of the United States Department of Agriculture, New Orleans, Louisiana, have been in commercial production since 1962. Precooked dehydrated sweet potato flakes however, develop "off flavor" and "off odor" during storage thus limiting storage life. Researchers have investigated the effects of time, temperature, and exposure to oxygen on the stability of dehydrated sweet potato flakes. Time and temperature of storage apparently have little effect on the palatability of the reconstituted product in the absence of oxygen, but detrimental changes occur rapidly at higher temperatures and increasing levels of oxygen. Several literature reports have

suggested that "off flavor" (determined by "off odor") is caused by oxidation products of the carotenes. Further work indicates that substances other than carotenes may be involved in the development of "off flavor" and that the carotenes may be merely sensitive indicators of the state of oxidation. One fraction recently implicated with the development of "off flavor" is a lipid fraction containing a small amount of carotenoid epoxide. This fraction appears to contain other compounds which may be the precursors of "off flavor." The major objective of this research was to isolate the compounds present in this lipid fraction and to determine the relationship of these compounds to "off flavor" in oxidized dehydrated sweet potato flakes.

CHAPTER II

REVIEW OF LITERATURE

Dehydrated Sweet Potatoes

The success of the white potato flakes and the decline in consumption of sweet potatoes led to the development of precooked, dehydrated sweet potato flakes. Early market research undertaken with groups of household and institutional users indicated a favorable reaction to sweet potato flakes because they were easy to prepare, saved time and labor, and added variety to menus. Consumer acceptance was good when the flakes were used in mashed sweet potatoes and in casseroles. Few consumers reported any difficulty in reconstituting the product or in following the recipes (1, 2).

Results of a later study with both a small trained panel and a large consumer panel indicated that color, flavor, and texture were good when 2 parts water to 1 part flakes by weight was used for reconstitution. This study indicated a lack of uniformity among samples from the same processor and from different processors. All of the reconstituted dehydrated sweet potato flakes were scored as slightly less than full-flavored. Panel members made many comments on the "off flavors" present in the samples (3).

The problem of "off flavor" in reconstituted dehydrated sweet potato flakes had long been recognized. Lambou had reported in 1956

that the storage temperature of the raw sweet potato had a profound influence on the palatability of the reconstituted dehydrated product (4). Reconstituted products made from raw sweet potatoes stored at 60 F, and from 70 to 75 F for 2 to 5 months were found palatable.

The original process of Deobald for making sweet potato flakes was applicable only to cured and stored sweet potatoes (5). A more appropriate method for use of freshly-harvested sweet potatoes involved the addition of limited amounts of alpha- and beta-amylase after cooking and pureeing to give the proper starch conversion required for flake production (5).

Vonesch, Ordonez, and Conti reported a weight loss in preserved sweet potatoes due to a decrease of starch and a change in water content (6). The content of soluble carbohydrate materials changed during preservation independently of the starch. Conversion of starch is brought about by the enzyme activation technique used in most commercial plants on fresh and cured sweet potatoes. This method involves rapidly heating the peeled and ground roots to a specified conversion temperature to allow naturally present amylases in the puree to act on the starch, then heating to above 200 F for cooking before dehydration in a double drum dryer (7).

Another major problem in the manufacture of dehydrated sweet potato flakes is a discoloration that masks the natural color. The high degree of unsaturation of the carotenoids makes them susceptible to oxidation with resulting color loss after the food containing

them has been dried (8). In a study conducted by Hoover sodium acid pyrophosphate was proven effective as a color preservative for sweet potato flakes (9). A 3 to 1 mixture of sodium acid pyrophosphate and tetrasodium pyrophosphate produced a somewhat better flavored flake with relatively little sacrifice in color preserving qualities.

Discoloration in sweet potato flakes may also be caused by gamma ray irradiation of the carotenoids (10, 11). This discoloration according to Lukton and Mackinney was apparently caused by secondary reactions and depends upon the extent to which free radicals or peroxides formed in the surrounding medium are available for reaction (11). These researchers found films of pure beta-carotene and lycopene in the solid state to be remarkably stable, even in the presence of air. Therefore, where destruction occured they believed it was initiated by products of other reactions, normally products found in the lipid fraction.

The major problem in the production of sweet potato flakes remains their short shelf life brought on by development of "off flavor" during storage. Georgia Red and Centennial varieties of sweet potatoes were studied under controlled storage at 15.5, 10, and 4.5 C (12). Boggess, et al. found that the fatty acid composition of the sweet potatoes changed during storage, especially at the low temperatures and theorized that synthetic and oxidative processes occurred in the fatty acids at the same time (12). They also suggested that such lipid changes were directly related to the organoleptic changes of storage. Deobald and McLemore recognized

that the cost of packaging precooked dehydrated sweet potato flakes in low oxygen atmospheres would delay or prohibit commercial production and attempted to eliminate this step with an antioxidant (Tenox VI to 0.1%) (13). The antioxidant action of this compound, however, could be demonstrated only at intermediate oxygen levels. The most stable flakes were those stored at 70 F in nitrogen containing 2% or less oxygen.

Purcell found it difficult to isolate and identify minor carotenoid pigments of plant tissue as they are often lost in the usual chromatographic procedures on columns sufficiently large to separate the major pigments (14). Purcell also found separation of carotenoids by adsorption chromatography was simplified if the carotenoids were first separated into classes according to their solubility in various solvents. In order to accomplish this Purcell used the procedure of Petracek and Zechmeister to verify the partition coefficients of the various pigments (15).

Purcell, investigating the major pigments in Goldrush variety sweet potatoes in raw, pureed, and precooked dehydrated flake products stored at both freezing and ambient temperatures reported that seven pigments constituted 98% of the total pigments (16). Beta-carotene made up 89.9% of the total, and no alpha-carotene was found. Results of this study indicated that the relative amounts of the individual carotenoids did not change appreciably during processing of the sweet potatoes into flakes or during storage of the flakes. Carotenoids were apparently not

destroyed by processing. Contrary to the conclusions drawn by Falconer, et al. in work with "off flavor" development in dehydrated carrots, Purcell and Deobald did not believe the results obtained offered a satisfactory explanation for the origin of the hay-like flavor and odor which develop in deteriorated sweet potato flakes (17). These results indicated that compounds associated with the carotenoids might be the major cause of the undesirable sensory changes in sweet potato flakes.

In a subsequent study Purcell and Walter studied the carotenoids of the Centennial variety sweet potato, the most commonly grown sweet potato in the Southeast since 1962, and reported that beta-carotene made up the largest part of the pigment, 86.35%. They also found 0.90% alpha-carotene (18).

In 1968 Purcell and Walter investigated autoxidation of carotene in food products using labeled beta-carotene (19). Results of the study indicated that the mechanism of autoxidation of carotene in food products was complex. Further studies are currently underway to determine the identity of labeled fractions which arise from beta-carotene in dehydrated sweet potato flakes. The 91% recovery of added label in this study suggests that volatile components may represent a significant part of carotene oxidation products.

Jones examined by sensory evaluation the relationship of carotenoids to "off odor" and "off flavor" development in dehydrated sweet potato flakes and reported that beta-carotene was not the precursor of the "off odor" and "off flavor" of precooked, dehydrated

sweet potato flakes (20). Additional results suggested that "off odor" and "off flavor" might be associated with some of the other oxidized carotenoid fractions or the non-saponifiable fraction of carotene.

Cox reported that the first three carotenoid fractions were not the precursors of "off odor" in precooked, dehydrated sweet potato flakes (21). Cox also found that "off odor" development in precooked dehydrated sweet potato, carrot and white potato flakes was apparently caused by different compounds peculiar to each product. Results obtained by Cox, however did indicate that compounds associated with the carotenoids, especially fraction 8, might be precursors of "off odor" in precooked dehydrated foods.

Sensory Evaluations

In order for a chemical to have an odor, it must vaporize and pass into the nasal cavity (22). It is not known whether the molecules dissolve in the fluid on the lining and the solution comes in contact with the olfactory cells or whether the hairs of the cells penetrate the mucous and come in contact with gas. There is a tremendous area for receiving odor stimuli; the varieties of odor stimuli requiring appraisal and identification can be numbered in the tens of thousands. Bedichek stated that this capacity for appraisal and identification of odor stimuli may reach to the hundreds of thousands in humans; in species more macrosmatic than man he feels the reach of the olfactory function is imponderable (23).

Amoore stated that the odor of a chemical is determined by the structure of the molecule, in particular by its size and shape (24). This theory would help account for the fact that chemicals with utterly unrelated empirical and structural formulae often exhibit very similar odors.

Amerine, Pangborn, and Roessler showed that some important components contributing to the aroma of foods have been isolated and identified, but researchers usually fail to establish which of the compounds isolated are responsible for specific sensory properties (25).

Later studies attempted to determine what particular combination of compounds makes an appealing odor or flavor (26). Much of this appeal is a matter of habit and conditioning, but exactly how much has not yet been established.

Boggs and Hanson reported that aspects of flavor might be judged by sniffing a food since flavor is considered a combination of odor and taste (27). Many researchers have reported better results with judgments of odor than with flavor. They have also reported that sensory fatigue set in more slowly with sniffing than with tasting.

Stone, Pangborn, and Ough reported that sniffing from bottles or beakers is the most widely used method of measuring odor intensity and quality (28). Sniffing is the most simple and economical of all procedures, but there are certain limitations which detract from its usefulness, chiefly the intensity of odor of the compound which is extremely hard to control.

Their report also indicated that rating of odor quality can lead to spurious results because preference and degree of liking influence a quality score. The researchers concluded it was better to rate the degree of difference in a specified odor characteristic between a treated and control sample, or between a treated and a commercial sample of known acceptance than to attempt to obtain scores of absolute quality (28).

In 1962 Stone, Ough, and Pangborn had demonstrated that odor difference thresholds could be established for panel members (29). These researchers found problems associated with training their panel to be minor.

In 1962 Arfmann and Chapanis reported that taste and odor thresholds were higher for smokers than for non-smokers (30). However, determination of a low threshold did not seem to be correlated to a subject's ability to act as an accurate judge.

A study by Bennett, Spahr, and Dodds showed that training of judges improved their performance in the evaluation of aroma and flavor (31). Mitchell stressed the necessity for concentration on the part of the judges (32, 33). The importance of psychological and physical conditions on the sensitivity of the taste difference test was emphasized. The human subject is not capable of machine-like performance, but is susceptible to influence by many physical and psychological conditions. Mitchell believed the laboratory supervisor would be better able to properly interpret results when the various influences that might affect his subjects on any particular test were recognized (33).

Raffensperger and Pilgrim found that knowledge of the stimulus variable was an aid in discrimination tests (34). The researcher should provide judges with enough information to sustain interest but not so much as to prejudice the panel (27).

Harries reported in carrying out sensory tests that there was a tendency for judges to choose the central sample of three as the odd one in the triangular tests (35). A similar phenomenon was found to exist in the "two-out-of-five" test. In the latter test such bias was reduced considerably by circular presentation.

Analytical Methods

Stahl has pointed out that taste panels adequately and properly carried out may be expensive. Instrumental methods of analysis, properly conceived, may be far less expensive, but useful only if significantly correlated to the sensory evaluation (36).

Mackay, Lang, and Berdick reported use of an ionization-type detector in the measurement of odors (37). Direct sampling techniques were evolved in which the same sample of air taken into the nose for sensory evaluation could be described by a pattern of constituents similar to a conventional gas chromatogram. Results were obtained from a variety of foods, beverages, and flavoring materials and were related to sensory experience. Ionization detectors are useful in such studies for their discriminating ability as well as their extremely high sensitivity.

The introduction and refinement of gas chromatography has provided the research scientist with his most powerful tool for determining volatile components. Appropriate tests with isolated fractions and compounds should help provide the answers to many questions concerned with the significance of the substances isolated (38).

Application of gas chromatography is now widespread. This technique can be used for analysis of mixtures of volatile or vaporizable compounds boiling at any temperature between absolute zero and 450 C, and for any substance which can be heated sufficiently without decomposition to give a vapor pressure of a few millimeters of mercury (39, 40). Gas chromatography has opened a way to investigate multicomponent, complex mixtures the analysis of which was once impossible or required the combination of several analytical methods. By means of a great number of adsorbents and liquid phases with diverse properties, it has become possible to investigate and analyze inorganic and organic compounds of all types (40).

In gas chromatographic analysis a solvent extract containing the substance to be determined is carried by a stream of inert gas into intimate contact with a thin film of non-volatile liquid spread on the surface of a powdered support (such as clay) inside a long tube or "column" (41). By a process similar in principle to fractional distillation, the mixture is resolved into its components in this column. Analysis is accomplished by observation and measurement of the response of a detector that can sense the presence of the con-

stituents (22, 41). Many factors other than the chemical nature of the absorbing compound influence the type of response of the detector. These factors include cell geometry, the temperature and pressure of the carrier gas, and the applied potential. All of the variables however, may be standardized, and appropriate electronic gadgetry can be designed to provide a signal that will operate a recorder (41).

At the present time any mixture whose constituents contain molecular weights in the range 2 to 400 and an appreciable vapor pressure, can be examined with a commercial mass spectrometer with a high probability that a successful analysis will be made. The sample required for analysis is small. The total time to make an analysis may be several minutes to several hours depending on the complexity of the sample, the information required, and the calibration data available. If no further calibration is needed, analysis is followed by computation which may require several minutes to several hours (42).

High speed computing is being used more and more in flavor identification studies to help reduce the computation time necessary, especially for routine and semi-routine analyses (42, 43). Gas chromatographs are now frequently coupled to mass spectrometers and, in turn, the output from the spectrometer is fed into a computer to make the tedious mass-number calculations (43). Gas chromatography and computer handling of data have not yet achieved the role desired in flavor evaluation because of the difficulty of correlating gas chromatographic peaks with constituents producing aroma.

CHAPTER III

EXPERIMENTAL PROCEDURES

Introduction

Following the work of Jones and Cox, raw Centennial variety sweet potatoes were extracted with acetone and hexane, and fractions 8 to 10 then were removed by column chromatography (20, 21). In efforts to secure purer samples than those used by Jones and Cox additional column chromatography and preparative thin layer chromatography were used before the samples were oxidized. Partition fractions from sweet potato lipids were also obtained and subjected to various methods of chromatography before oxidation. Pure compounds were still not obtained. Later panel evaluation of the oxidized samples in comparison to good (stored in nitrogen) and "poor" (stored in oxygen) reconstituted sweet potato flakes showed much confusion among the judges and little correlation between the odor of the samples and the odor of the reconstituted flakes.

Another approach was then tried using a steam distillate prepared from reconstituted sweet potato flakes for comparison with reconstituted flakes. This method also produced impure compounds which tended to confuse the judges during sensory evaluation. The next approach to the problem involved the use of a short path molecular still to prepare distillate samples at temperatures lower than those needed for steam distillation. All distillate samples were

subjected to chromatographic analysis (column, thin layer, preparative and/or gas) before oxidation and presentation to a panel for evaluation. Some samples were saponified before distillation; others were not. Samples were prepared from raw and cooked Centennial variety sweet potatoes. No pure compounds were obtained by any of the analytical methods employed.

After oxidation various samples were presented to panels for sensory evaluation. Statistical analyses were performed on sensory panel data from studies which indicated some similarity between the oxidized samples and the odor of the reconstituted flakes.

Due to the inability of the judges to relate the odor of the oxidized samples to the odor of reconstituted sweet potato flakes throughout this study, a final sensory evaluation was carried out using untrained students in an undergraduate experimental foods class as judges. Seasoned samples of good and "poor" reconstituted flakes were presented to the panel in a triangle test to determine if persons unfamiliar with sweet potatoes could identify the "poor" samples; and if they could do so, whether they would voice a strong dislike toward these "poor" samples.

A detailed description of the experimental procedures used follows.

Procedures

Late in June, 1968, approximately 1 kg of raw Centennial variety sweet potatoes was peeled, eyed, diced, and pureed in a 1 gallon blender with water and methanol. The puree was filtered

through a Buchner funnel, and the mat was extracted with equal volumes of acetone and hexane to remove the carotene and other lipids. The extracts were combined in a separatory funnel until distinct layers were formed. The bottom layer was then passed through ether. The acetone was washed out with water and the extract was dried with sodium sulfate and evaporated in a rotary film evaporator to about 100 ml. The sample was placed on a magnesium oxide--Hyflo Supercel column (18 x 350 mm) with hexane and developed with hexane and increasing amounts of acetone (2 to 10%) to separate the various fractions.

Fractions 8 to 10 on the column were removed and placed on a small column (8 x 150 mm). Seven distinct bands were seen and each was removed separately. Each of these 7 fractions was rechromatographed through a column of alumina activated at 350 C and deactivated with 10% water. Fractions were placed on the columns with hexane and developed with 5 to 10% ether in hexane. Separate bands in each fraction were collected, evaporated, taken up in 2 ml of hexane, and stored at -10 C in tightly covered vials until spotted on thin layer chromatography (TLC) plates.

TLC plates were made by suspending 30 g silica gel G in 60 to 65 ml distilled water and streaking the slurry in a 0.250 mm layer. Plates were dried overnight at room temperature and then activated at 120 C for 1 hour. Two amounts of each sample were placed on the plates, 0.050 and 0.200 ml. Plates were developed in benzene, dried, and examined under ultraviolet light. Fluorescent spots were circled

in pencil. Plates were then sprayed with sulfuric acid and placed in an oven for charring at 120 C. Solvent remaining in samples after TLC was evaporated in air, the vials were tightly covered and stored at room temperature in the dark to determine what "off odors" would develop during storage. After development and charring of the plates, relative frontal movement (R_f) was calculated for each spot.

Five judges were selected as a preliminary panel. The judges were trained before the actual gathering of data began. At this time the judges were given information about the study and about the type of testing to be done. The principles of sensory testing were briefly explained, as well as the workings of the score cards to be used.

Ten of the 39 isolated fractions were judged by the researcher to have a definite odor. These were presented to a panel for comparison with samples of good (stored in nitrogen) and "poor" (stored in oxygen) reconstituted sweet potato flakes. The previous experience of Jones and Cox indicated that panel results were more consistent when the sweet potato flakes were reconstituted than when they were in the dry state (20, 21). To help prevent the problem of olfactory fatigue, 5 samples were used at a time in comparison with the odor of the good and "poor" reconstituted flakes. Samples were arranged on trays in random order for judging. Each judge was asked to evaluate each series 3 times. Judges were asked to make comments about the samples when they felt such comments were necessary. The comment made most often was that the judge could detect no odor in the sample being tested. This comment increased as the study was continued indicating the strength of the samples decreased with time.

Specific times for judging were set up according to the schedule of the judges. Judging was done between 10:00 and 11:00 A.M. daily, Monday through Friday. One-half cup of each type of sweet potato flake was reconstituted with one-half cup of boiling water for each test. Three to 4 minutes after reconstituting, the sweet potatoes were placed in small beakers for sniffing. Beakers were not covered for the first series of judgings; for the second and third series foil covers were provided. Judges were asked to sniff one vial, compare it to the odor of the flakes in the beaker on the same tray and report samples as being alike, similar, or different. Instructions given to judges and preliminary score cards are shown in Appendix A. The sweet potato flakes used in this study were Goldrush Variety, Run No. 169-1, stored in air and stored in nitrogen, a research product of the Southern Regional Research Laboratory at New Orleans, Louisiana.

In addition to the 10 compounds from fractions 8 to 10, the same panel was asked to evaluate 9 fractions prepared by partitioning sweet potato lipids in comparison with good and "poor" reconstituted sweet potato flakes. These fractions had been prepared by placing the crude carotene solution in a counter current apparatus and partitioning it between 95% methanol and hexane. Tubes 1 through 5, 6 through 10, 11 through 15, 16 through 20, 21 through 25, 26 through 30, 31 through 35, 36 through 40, and 41 through 50 were combined. The solvent was evaporated in a rotary film evaporator and the samples were taken up in 2 ml of hexane and placed in vials. The

hexane was evaporated in air; the vials were tightly covered and stored at room temperature in the dark until judged by the panel. All fractions developed a definite odor during storage.

A compilation of the judges' scores for the chromatographed fractions and the partition fractions is shown in Table 1 in Appendix B. No statistical analysis was performed on these preliminary data.

In late January, 1969, a second set of fractions was prepared; this time starting with 2.5 kg of raw Centennial variety sweet potatoes. Fractions 1 through 8 were removed from the column before it was allowed to go dry. The column was then carved out to separate fractions 9 through 13. Fractions 8, 11, and 13 were rechromatographed as before on magnesium oxide--Hyflo Supercel columns. Fraction 8 yielded 6 distinct bands, fraction 11, 4 bands, and fraction 13, 6 bands. Each band from fraction 8 was placed on an alumina (10% water) column with hexane and developed with 5% ether in hexane. The bands from fractions 11 and 13 were not further chromatographed.

Partition fractions were prepared in a counter current apparatus. Tubes 1 through 15, 16 through 23, 26 through 40, and 41 through 50 were combined. All fractions were washed with distilled water in separatory funnels and passed through ether. Partition fractions were evaporated in a rotary film evaporator to about 100 ml and placed on a magnesium oxide--Hyflo Supercel column. The columns were developed with 5% acetone in hexane. Fraction 1 - 15 yielded 1 band, fraction 16 - 23, 3 bands, fraction 26 - 40, 5 bands, and fraction 41 - 50, 1 band.

The chromatographic bands were carved out and eluted from the adsorbent with acetone and hexane. The solvent was evaporated from the chromatographic and partition fractions in a rotary film evaporator. The samples were taken up in 2 ml of hexane and placed in vials. After the hexane was allowed to evaporate in air, the vials were tightly covered and stored at room temperature in the dark until oxidized "off odors" developed.

After oxidation of the fractions, the same preliminary panel was asked to sniff all of the vials to determine which fractions had developed distinct odors. Only 3 of the 37 fractions were eliminated as having no odor.

These fractions, as was done previously, were presented to the judges to be compared to reconstituted sweet potato flakes which had been allowed to develop "off flavor." This time the reconstituted flakes were placed in vials of the same size and shape as those in which the fractions were presented because in the previous panel the judges seemed to be misled by the intensity of aroma coming from the flakes. The use of vials for the reconstituted flakes was helpful in alleviating this problem.

Judges were asked to evaluate samples as being alike or not alike. A revised score card as shown in Appendix A was used in this second preliminary study. An analysis of variance was determined on the data obtained from these tests.

Both preliminary studies were conducted in an experimental foods laboratory at the University of North Carolina at Greensboro

in an atmosphere in which there was little control over extraneous odors. The remaining studies were carried out in the Food Science Department at North Carolina State University at Raleigh using facilities specifically designed for sensory evaluations.

A steam distillate prepared from reconstituted sweet potato flakes by the method of Walter, et al. was found to develop an odor similar to that of the "off flavored" sweet potato flakes (44). A steam distillate was then prepared to compare to the reconstituted sweet potato flakes in addition to the carotene fractions.

In June, 1969, approximately 1500 g of peeled Centennial variety sweet potatoes was prepared in a manner previously described and dried on a drum drier. The flakes were placed in an amber jar, the jar was evacuated, flushed with nitrogen, tightly covered and stored overnight.

Two 70 g portions of the sweet potato flakes were reconstituted and steam distilled. The distillates were combined and extracted with distilled ether. The ether was evaporated in the rotary film evaporator, the distillate was tightly covered, and stored under nitrogen in the refrigerator.

Purified ethyl ether was prepared by stirring 2 l of ethyl ether with 400 ml of 10% sulfuric acid containing 40 g of ferrous sulfate in a large Erlenmeyer flask with a magnetic stirrer for 1 hour. The ether was decanted into a distillation flask and distilled slowly (1 to 3 ml per minute). Approximately 80% of the treated ether was collected, and the balance was discarded.

Carotene fractions 8, 9 to 12, and 13 were prepared as previously indicated from a magnesium oxide column. After evaporation all samples were covered and stored at -10 C until needed.

The fractions and the steam distillate were removed from cold storage. Hexane was added to each sample. Five ml from each fraction was placed in each of 5 vials. The remainder of each fraction was stored under nitrogen at -10 C. Five ml from the steam distillate was placed in each of 10 vials. The remainder of the distillate was also stored under nitrogen at -10 C.

The solvent in all vials was evaporated in air. Five of the vials containing the steam distillate were placed in a dessicater and a vacuum was drawn. The vacuum was relieved with nitrogen and the vials were tightly covered and stored at room temperature in the dark until evaluation by a panel of judges. The remaining vials were treated in the same manner before storage except that they were placed in a vacuum which was relieved with oxygen.

A panel consisting of 15 individuals was asked to compare odors in the vials to those of good and "poor" reconstituted sweet potato flakes and report the sample as being alike or different. This panel, as well as all of the subsequent sensory panels, was composed of graduate students, technicians, secretaries, and faculty members in the Food Science Department at North Carolina State University at Raleigh. All panel members had done previous sensory testing, so no preliminary training was given to the judges. The second revised score card shown in Appendix A was used in this series and throughout

the remainder of the study. Judges were asked to compare the odors of the samples to the odors of good and "poor" reconstituted flakes at each session. One cc of reconstituted sweet potato flakes was placed in each vial using a 30 cc syringe. Good and "poor" flakes were randomly labeled as X or Y and presented in random order. Each judge was asked to compare each vial to the X or Y sample in three separate trials. All evaluations were carried out in the booths for sensory evaluation in the Food Science Department. Deep red filters were used in the booth lights. Results of this series are shown in Table 2 in Appendix B. No statistical analysis was performed on these data since no sample was scored as definitely like the good or "poor" sweet potato flakes.

A second steam distillate was prepared in September, 1969, and stored at -10 C. The slurry which had been steam distilled was mixed with an equal volume of methanol, covered, and stored in the refrigerator. Upon removal from storage the slurry was extracted with 50% acetone in hexane as previously described. After passage through ether and removal of the water, the sample was saponified with potassium hydroxide before drying and evaporation in the rotary film evaporator. The sample was cloudy when taken up in hexane; therefore, it was centrifuged at 17,000 RPM for 10 minutes at 0 C before placing on a magnesium oxide column. Bands 8, 9 to 12, and 13 were removed and prepared for subsequent analysis. Vials in this series, and all further series were coded with a 3 digit number before presentation to the panel.

"Poor" sweet potato flakes were obtained by opening cans of sweet potato flakes which had been stored in air and allowing further oxidation to occur for 3 days before the start of the panel. This procedure was followed in an effort to produce a more pronounced oxidation than had been present in previous "poor" samples.

The sensory panel was conducted as before. Sixteen judges evaluated each series 3 times. Due to changes in personnel, the panel was not composed entirely of the same judges as the former panel. The results of this series are shown in Table 3 in Appendix B. No statistical analysis was performed on these data again because no fraction was scored as definitely like the reconstituted sweet potato flakes.

In October, 1969, a steam distillate was prepared from sweet potato flakes starting with approximately 750 g of peeled, raw Centennial variety sweet potatoes. Ten ml hexane was placed over the evaporated sample. Two ml of the sample was placed on an alumina (5% water) column. The remainder of the distillate in hexane was stored under nitrogen at -10 C. The column was developed with 350 ml of 5% ether in hexane, 100 ml 50% ether in hexane, 200 ml 5% methanol in ether, 200 ml 50% methanol in ether, and finally 400 ml of methanol. The eluate was collected in 10 ml aliquots using an automatic fraction collector.

TLC plates were prepared and were spotted with 0.200 ml from every third tube from the fraction collector. The plates were developed in benzene, dried, and examined under ultraviolet light.

Plates 1 and 2 were sprayed with sulfuric acid and potassium dichromate solution and placed in the oven for charring. The remaining plates were treated in the same way except that they were sprayed with 0.5 g potassium permanganate dissolved in 15 ml concentrated sulfuric acid. Four plates were spotted with samples from the chromatographed distillate. Plates 1 and 4 showed no migrating spots. Movement of the spots was measured, and the R_f values calculated for spot movement shown on plates 2 and 3.

One-fifth of fraction 8 obtained from the steam distilled sweet potato flakes was chromatographed on alumina (5% water) and developed in the same manner as the steam distillate. In addition a blank was prepared by evaporating 250 ml of 1:1 acetone and hexane combined with 25 g of magnesium oxide--Hyflo Supercel and filtered. One-fifth of the blank was chromatographed on alumina (5% water).

Fraction 8 and the blank were spotted on TLC plates starting with tube 24 and continuing through tube 75. Plate 8A was spotted with 0.200 ml of hexane. R_f values of fractions were calculated.

In an attempt to determine if a combination of lipid and sweet potato volatiles oxidized to give hay-like "off odor," 5 ml of steam distillate and 5 ml of fraction 8 were combined in a vial, and the solvent was evaporated in air. The initial product had some of the characteristic odor of the sweet potato, but the oxidized product did not have the definite hay-like "off odor" of oxidized sweet potato flakes.

Two ml of steam distillate was again chromatographed on an alumina (5% water) column, and the eluate was collected in a fraction collector set to collect 10 ml aliquots. After checking the movement shown on TLC plates 2 and 3, tubes 1 through 35, 36 through 54, and 55 through 113 were combined, the solvent evaporated, and the samples stored under nitrogen at -10 C.

Two ml of fraction 8 was also chromatographed on alumina (5% water). The movement shown on TLC plates 5 and 6 was checked before tubes 1 through 35, 36 through 64, 65 through 75, and 76 through 113 were combined. The solvent was evaporated and the samples were stored under nitrogen at -10 C.

Plates were prepared for preparative chromatography by suspending 60 g silica gel G in 120 ml distilled water; plate thickness was 0.750 mm.

Infrared spectograms were obtained for the 3 samples of steam distillate and 4 samples of fraction 8 in carbon tetrachloride. The presence of C=C could not be verified by infrared spectroscopy because the sample size apparently was too small to show weak bands. The C=O bonds shown were too small to be conclusive, thus indicating that carbonyls were minor constituents of these fractions.

The carbon tetrachloride was evaporated from the above samples, and the samples were taken up in hexane and streaked on plates prepared for preparative chromatography. All plates were developed in benzene, examined under ultraviolet light, and the edges sprayed with potassium permanganate in sulfuric acid. Each band was scraped

off the plate, placed in 10% ethanol in ether, and filtered. The solvent was evaporated, and the sample transferred in hexane to a vial and stored under nitrogen at -10 C.

Preparative plate 1 was streaked (using a streaker) with 0.250 ml of steam distillate from tubes 36 through 54. Six bands were observed at 23-33 mm, 43-58 mm, 103-125 mm, 125-142 mm, 161-172 mm, and 172-187 mm. Preparative plate 2 was streaked with 0.50 ml of steam distillate from tubes 1 through 35. Bands were observed at 18-28 mm, 37-56 mm, and 137-191 mm. Plate 3 was streaked with 0.50 ml of steam distillate from tubes 55 through 113. Bands were observed at 31-48 mm, 101-112 mm, 122-135 mm, and 163-180 mm.

Preparative plate 4 was streaked with 0.50 ml of fraction 8 from tubes 1 through 35. A single band slanting from 105-148 mm on the right to 150-182 mm on the left was observed on this plate. Plate 5 was streaked with 0.50 ml of fraction 8 from tubes 36 through 64. Bands were observed at 43-52 mm, 130-153 mm, and 180-193 mm. Plate 6 was streaked with 0.75 ml of fraction 8 from tubes 65 through 75. Bands on plate 6 were observed at 33-45 mm, 121-140 mm, and 147-177 mm. Plate 7 was streaked with 0.50 ml of fraction 8 from tubes 76 through 113. A single band at 172-192 was observed on this plate.

After all preparative chromatography was completed, all vials were removed from -10 C storage. The samples were dried in air, covered, and stored in an oxygen-rich atmosphere.

In December, 1969, approximately 750 g of raw Centennial variety sweet potatoes was made into flakes in preparation for steam distillation. Since the steam distillate contained moisture after evaporation, attempts were made to remove this moisture by taking the sample up in ethanol, adding distilled ether, drying with sodium sulfate for 2 hours, and evaporating the solvent on the rotary film evaporator. The moisture was still present so the sample was taken up in ethanol and distilled ether again, washed with distilled water in separatory funnels, dried with sodium sulfate, and the solvent evaporated. Some moisture remained in the distillate sample, but no further attempts were made to remove it. The odor of the steam distillate had changed during the attempts to remove the moisture from the sample indicating that some of the volatiles were removed even though the moisture remained. The sample was stored under nitrogen at -10 C.

The slurry from the steam distilled sweet potatoes was extracted and chromatographed on a magnesium oxide--Hyflo Supercel column. Fraction 8 and the bands immediately above and below it were carved out, extracted, and stored. No further work was done with the slurry.

Hexane was added to each of the 4 sub-fractions of fraction 8 used in the preparative chromatography in November, 1969. Hexane was added to the steam distillate prepared in October, 1969 and December, 1969. One ml from each sub-fraction of fraction 8 was combined with a 1 ml aliquot from each of the steam distillates in vials. The

solvent in the 8 vials was evaporated in air, and the vials were covered and set aside to oxidize. These samples did not develop strong enough odors for use by a sensory panel.

Vials prepared from preparative chromatography bands were removed from storage in the oxygen-rich atmosphere and sniffed by the researcher. Odors were detected in all but 2 of the 21 vials, but some of the odors were very faint. Odors were described as somewhat oily, musty, stale, slightly terpenoid, medicinal or somewhat fruit-like. After sniffing, all vials were flushed with nitrogen and returned to -10 C storage. Since many of these odors were not very distinct, and few resembled the "off flavored" sweet potato flakes, this set of vials was not used with a sensory panel.

In December, 1969, short path molecular distillation was started. Pictures of the short path still used are shown in Figures 1, 2, and 3. Total lipids were extracted from 3 g of raw freeze dried Centennial variety sweet potatoes. A concentrated hexane solution of the lipids was placed in the still, and the hexane was evaporated with a stream of nitrogen. The top cavity of the still was cooled with dry ice, and the still was connected to a receiver flask cooled with dry ice, acetone, and a vacuum source. When the pressure had been reduced to about 1 mm, as indicated by the sound of the vacuum pump, the bottom of the still was heated with boiling water for 1 minute. After an additional 2 minutes the vacuum was broken. Ten ml of hexane was added to the still, and the dry ice was removed from the top cavity. The hexane was distilled into the

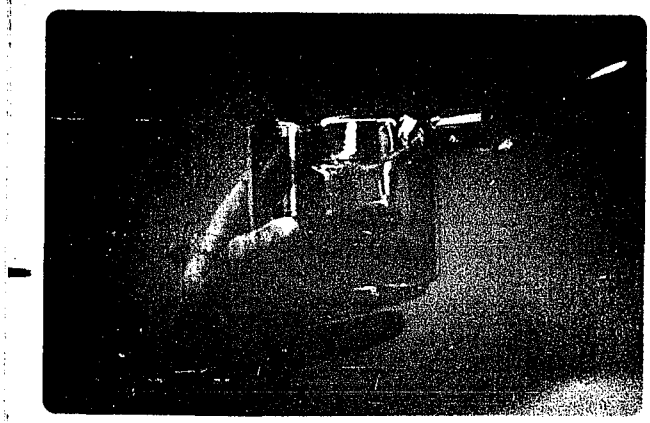

Figure 1

Filling the Short Path

Molecular Still.

Figure 2

Exhausting the Still

with Nitrogen.

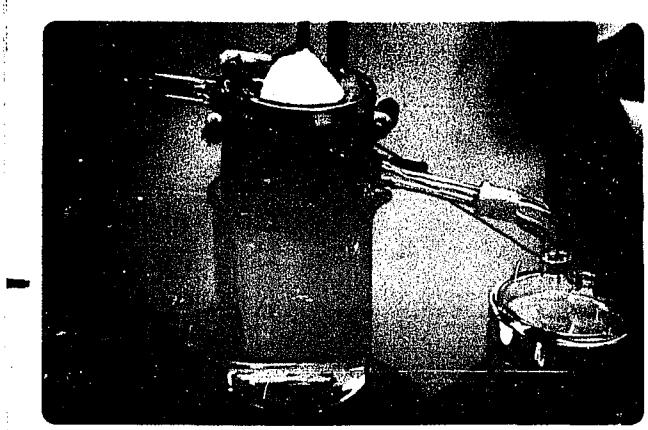

Figure 3

Distillation in Process.

receiver flask carrying with it the distillate which had been trapped on the top plate of the still. The non-volatile residue was chromatographed on a magnesium oxide--Hyflo Supercel column, developed, and carved out.

Distillates and chromatographic fractions were evaporated to 20 ml, and 0.005, 0.020, and 0.050 ml of each was spotted on TLC plates for developing. The spots on the plates were very small. It was estimated that 4 to 5 times as much of the distillates and 2 times as much of the non-distilled samples should have been used in spotting the plates. The distillates and chromatographic fractions were concentrated to 1 ml, and 0.10 ml distillates and 0.050 ml non-distillates were placed on TLC plates. Plates were developed in naptha (70 ml), ether (30 ml),and acetic acid (2 ml). Spot movements were measured and the R_f values were calculated.

Carotene extracts of several batches of flakes and freeze dried sweet potatoes were combined and concentrated to 500 ml. No temperature above 30 C was used in this process.

Two hundred fifty ml of the above concentrate was saponified with potassium hydroxide. Eighty ml of the saponified concentrate was distilled in the short path still to produce the saponified unchromatographed distillate. One hundred seventy ml of the saponified concentrate was chromatographed on magnesium oxide--Hyflo Supercel. Fraction 8 was carved out and extracted. One-half of this extract received no further treatment. The remaining half of the extract was distilled in the short path molecular still to produce saponified chromatographed distillate and a saponified fraction 8 non-distillate.

All samples, plus a beta-ionone standard and aliquots of chromatographic fraction 8 before distillation, were spotted on TLC plates and developed. Spot movements were measured for all samples which showed migrating spots, and R_f values were calculated.

In December, 1969, 3 kg of raw Centennial variety sweet potatoes was extracted. One-half of the extract was saponified with potassium hydroxide. Half of each sample was placed in the short path still, and the volatiles were trapped. The non-volatiles of the saponified distillate were chromatographed on magnesium oxide--Hyflo Supercel. Those of the unsaponified distillate were spilled. The fraction corresponding to 8 was carved out and extracted. The other half of each fraction was chromatographed on magnesium oxide--Hyflo Supercel, and fraction 8 was obtained.

On January 6, 1970, all samples were spotted on TLC plates, developed in naptha, ether, and acetic acid, sprayed with chromic acid spray, and charred in a 150 C oven. Spot movements were measured and R_f values calculated.

One tenth ml was removed from each of the 5 samples, placed in vials, and evaporated in air. One-tenth ml of benzene and 1 drop of 2, 4, dinitrophenylhydrazine solution (5% in trichloracetic acid) was added to each vial. The following day 0.10 ml of hexane was added to each vial and 0.05 ml of each sample was spotted on a TLC plate. The plate was developed, sprayed and charred. Spot movements were measured and R_f values calculated.

One one-hundredth ml of each sample was placed in a gas chromatograph. Part of the saponified distillate and saponified distilled fraction 8 were combined in a vial. Solvents in all vials were evaporated; the final drying was with nitrogen. All vials were then blown with oxygen, covered, and stored in the dark in an oxygen-rich atmosphere.

On January 15, 1970, the vials were removed from storage and sniffed to determine if "off odors" were present. All vials had developed a definite odor. Since the raw distillate, raw non-distillate, cooked distillate, and cooked non-distillate did not have odors which were similar to those of the sweet potato, they were not used in a sensory evaluation with a panel. The cooked saponified and unsaponified samples had odors which this researcher felt resembled those of "off flavored" sweet potato flakes. Vials containing these samples were covered, coded, encased in foil, and replaced in an oxygen-rich atmosphere until use by a panel of judges.

A panel of 10 judges was asked to evaluate the oxidized saponified and unsaponified samples in comparison with the odor of reconstituted "poor" sweet potato flakes. The reconstituted flakes were labeled X or Y at random. The saponified samples were presented for comparison one day and the unsaponified samples the next to avoid olfactory fatigue. Each judge was asked to evaluate each set in this series 3 times. It was not possible for all judges to be present at each session. A compilation of the judges' scores for this series appears in Table 4, Appendix B. No statistical analysis was performed on these data.

A gas chromatographic analysis of the head space of the flakes and samples used in the January, 1970 sensory evaluations was attempted, but the results were inconclusive.

On January 28, 1970, the vials containing oxidized samples of raw saponified and unsaponified fraction 8 and steam distillate and the combination of saponified distilled fraction 8 and saponified distillate were removed from storage. All 6 vials contained a definite odor. Vials were then encased in foil, coded, and returned to an oxygen-rich atmosphere until use by a panel.

Ten judges were again asked to evaluate the samples in comparison with the odor of the reconstituted "poor" sweet potato flakes. The panel was conducted with the saponified samples being presented for comparison one day while the non-saponified samples and the combination were presented the next day. The saponified samples were evaluated by the panel three times. The non-saponified samples and the combination were evaluated only twice before the samples deteriorated. As in the previous series not all judges could be present at all sessions. The results of this series are found in Table 5, Appendix B.

On January 28, 1970,the carotene extracted from cooked sweet potato flakes was saponified with potassium hydroxide and chromatographed on magnesia. Fraction 8 was carved out, extracted and preparative chromatography plates were prepared. Plate 1 was streaked with 0.050 ml of fraction 8, plate 2 with 0.10 ml, and plate 3 with 0.075 ml. The plates were developed and R_f values

were calculated from spot movement measurements. Plates were examined under ultraviolet light. Four flourescent bands were removed from the plates before the edges were sprayed with dichromate solution and charred in a 135 C oven. Bands were scraped off plates, treated as before, and stored in covered vials in an oxygen-rich atmosphere. No distinct odors developed as the fractions oxidized.

One hundred ml of hexane was added to the remainder of fraction 8. The sample was placed in a separatory funnel with 100 ml of 95% methanol and partitioned between the methanol and hexane to drive 1 hydroxy compounds and short chain alcohols into the methanol. After partitioning the methanol phase was extracted with ether. Addition of ether produced a single phase. Water was added to re-establish 2 phases and the water layer was discarded. After partitioning the phases were dried, and the samples taken up in 2 ml of hexane when the solvents had been allowed to evaporate.

The epi-phase and the hypo-phase were spotted on TLC plates in an attempt to separate alcohols from less polar compounds. Plates were developed, examined, sprayed, and charred. Spot movements were measured and R_f values were calculated. The remaining epi-phase and hypo-phase were placed in vials in hexane. The hexane was evaporated in air, and the vials were stored in an oxygen-rich atmosphere. No distinct odor was developed by either phase during oxidation.

On February 23, 1970, 2 kg of raw Centennial variety sweet potatoes was prepared and extracted. After evaporation to about 100 ml, the extract was subjected to short path molecular distillation. The slurry from the distillation was discarded.

When the distillation was complete, the distillate was subjected to gas chromatographic (GC) analysis in a Packard 7300/7400 series instrument with flame ionization detectors and a model 844 electrometer. Nitrogen was the carrier gas (25 ml per minute) on a 1/8 inch, 6 foot glass column with 10% OVI on Chromasorb G, 60-80 mesh. For the first analysis the GC was programed with an initial temperature of 50 C, an initial hold of 20 minutes, a programed temperature rise of 5° per minute to a final temperature of 200 C, and a final hold of 20 minutes. An aliquot of 0.01 ml of sample or standard was injected for each GC analysis.

A second analysis of the distillate was performed with an initial temperature of 30 C, an initial hold of 5 minutes, a programed temperature rise of 10° per minute to a final temperature of 200 C, and a final hold of 40 minutes. The third analysis was performed with an initial temperature of 90 C, an initial hold of 10 minutes, a programed temperature rise of 3° per minute to a final temperature of 200 C, and a final hold of 20 minutes. The GC program developed for the third analysis was used throughout the remainder of the study except for some of the head space analyses. The electrometer range was varied because of variations in the concentration of the samples.

GC analysis was carried out on 4 standards for comparison with graphs obtained from analysis of the distillate. The standards used were 0.01% beta-ionone, 0.10% isoeugenol, 0.10% citral, and 0.10% linalool.

On March 9, 1970, 2 kg of raw Centennial variety sweet potatoes was prepared and extracted. After evaporation to about 100 ml, the carotene crystals were removed by filtration before the extract was subjected to short path molecular distillation. The distillate obtained was divided into 1/3 and 2/3 portions. The larger portion was partitioned between 95% methanol and hexane to obtain the epi-phase and hypo-phase. GC analysis was performed on each phase and the whole distillate.

The slurry from the distillation was placed on a magnesium oxide--Hyflo Supercel column. The column was developed by the procedure previously outlined but the compounds present remained on the column. Apparently, the treatment during the short path molecular distillation had caused a polymerization of the compounds and these compounds could not be separated by column chromatography. This slurry was discarded.

Samples were prepared for the next sensory panel by adding hexane to the whole distillate and to the epi- and hypo-phases. Vials were encased in foil and coded; 3 vials were prepared for each sample. A 3 ml aliquot was placed in each vial and the solvent was evaporated in air. Oxygen was blown over each sample and the vials were covered and stored in the dark.

On March 7, 1970, while the samples were oxidizing, 2 kg of raw Centennial variety sweet potatoes was prepared and extracted. Carotene crystals were removed by filtration after evaporation to about 100 ml before the material was subjected to short path molecular

distillation. A GC analysis of the distillate was made. The distillate was saponified with potassium hydroxide and divided into saponified and non-saponified fractions. The saponified fraction was divided into acid and non-acid sub-fractions. The non-saponified fraction was partitioned between 95% methanol and hexane to separate the epi-phase from the hypo-phase. GC analyses were run on all samples at 1×10^{-10} or 3×10^{-11} electrometer sensitivity. After GC analysis the samples were taken up in hexane and placed in vials. After the solvent was allowed to evaporate in air, oxygen was blown over each sample. The vials were covered and stored in the dark.

A panel of 15 judges was asked to compare the odors of the oxidized distillate, epi-phase and hypo-phase, plus good reconstituted sweet potato flakes, and a blank to the odor of reconstituted "poor" sweet potato flakes. The blank was made by mixing 10 ml of agar in 30 ml boiling water and adding Hyflo Supercel and crushed potassium dichromate for color and consistency. Each judge evaluated each series 3 times and statistical analysis was performed on the data obtained.

GC analysis was performed on head space from samples and "poor" sweet potato flakes at the conclusion of the panel. Vials and syringes were warmed before injection into the GC. No distinct peaks were found by injecting 2 cc; this was increased to 10 cc in an attempt to place more material on the column. The initial temperature was 40 C, the initial hold was 10 minutes, the programed temperature rise was 3° per minute to a final temperature of 150 C, and a final hold of

10 minutes. An electrometer sensitivity of 3×10^{-12} was used for all samples. The results of the head space analysis were inconclusive. After completion of GC analysis the vials were stored at -10 C.

On April 13, 1970, a distillate was prepared in the short path molecular still from 2 kg raw Centennial variety sweet potatoes as previously described. This distillate was prepared to send to the International Flavors and Fragrances Company at Union Beach, New Jersey for mass spectral analysis. GC analysis was performed on the distillate as previously described at an electrometer sensitivity of 3×10^{-10}. After analysis the distillate was cooled with dry ice, blown with nitrogen, and sealed into an ampule for shipping. GC analysis was also performed on a standard containing 0.050 mg beta-ionone, 0.050 mg citral, and 0.050 mg linalool in a 50 ml solution. The graphs obtained in the GC analyses were sent with the distillate to IFF.

The vials used by the previous panel were removed from -10 C, recoded, and 1 cc of reconstituted good sweet potato flakes was added to each oxidized sample. Nothing was added to the vials already containing reconstituted good sweet potato flakes. A panel of 15 was asked to compare the odors in these vials to the odor of reconstituted "poor" sweet potato flakes. Each judge evaluated each series 3 times. A statistical analysis was performed on the data obtained. At the conclusion of this panel, the vials were again stored at -10 C.

The final sensory panel at Raleigh evaluated this same set of vials which was removed from -10 C storage for each judging session.

Vials were not recoded, but a sample of reconstituted commercially prepared good sweet potato flakes was added to the series of vials. A panel of 16 judges compared the odors of the 3 oxidized samples plus good sweet potatoes and the 2 samples of reconstituted sweet potato flakes to the odor of reconstituted "off flavored" flakes which contained no sugar. Each judge evaluated each set of vials 3 times. A statistical analysis was performed on the data obtained. Least significant difference (LSD) values were calculated to give an indication of the difference required between two treatment means to show significance.

In January, 1971 seasoned samples of reconstituted good and "poor" sweet potato flakes were presented to an undergraduate experimental foods class of 9 individuals at The State University College at Oneonta, New York. All samples were reconstituted using 3/4 cup of freshly boiled water to 3/4 cup of flakes. One tablespoon of butter and 1/4 teaspoon of salt were beaten into each sample. Samples were reconstituted at least 15 minutes before testing and were stirred periodically to assure a smooth product during judging. Samples were presented as a triangle test in random order to determine if these untrained students, generally unaccustomed to eating sweet potatoes, could identify the poor product. Each student performed the triangle test 3 times under conditions similar to a home situation. Students were asked to react to the texture of the samples as well as to the flavor. The score card used by this panel is shown in Appendix A.

CHAPTER IV

RESULTS AND DISCUSSION

In setting up this study, it was hypothesized that further fractionation of the lipid fractions implicated by Jones and Cox in the development of "off flavor" in dehydrated sweet potato flakes would isolate the compounds responsible for this "off flavor" (20, 21). When further separation was carried out by column chromatography, the fractions were found to contain vast amounts of non-carotenoid substances. Attempts were made to further purify the fractions by means of additional chromatography but these attempts resulted in the loss of all resemblance to "off flavor."

In the first preliminary study the 39 isolated chromatographic fractions showed marked tendency to blur and streak when spotted on TLC plates. Relative frontal movement (R_f) was calculated for each spot. These R_f values showed that a great many compounds were present in the fractions and indicated that many of the compounds were present in more than one fraction thus suggesting that complete separation of compounds had not been obtained on magnesia, alumina, or silica.

When the 10 oxidized fractions which developed distinct odors were presented to the preliminary panel, none was scored as being definitely like the good or "poor" sweet potato samples. The same results were obtained when this panel compared the odor of 9 partition

fractions to the sweet potato samples. No statistical analysis was performed on this preliminary study, but a compilation of the scores of the judges is shown in Table 1, Appendix B.

In the first preliminary study, the panel seemed somewhat confused by the intensity of the odors of the reconstituted flakes in the small beakers. In an attempt to overcome this difficulty, the sweet potato flakes were placed in vials of the same size and shape as those used for the samples in all other sensory evaluations. Use of the vials was helpful, but intensity of the odors of the samples and reconstituted flakes was a problem throughout this entire research project.

The score card (Appendix A) used in the second preliminary evaluation was revised to aid in the statistical analysis. This score card gave the judges more choices than had the first one. Samples were compared only with reconstituted "poor" sweet potato flakes in this set of evaluations.

Statistical analysis of the second preliminary study showed that again none of the samples was evaluated as definitely like the reconstituted "poor" sweet potato flakes. In replication one, 29.4% of the samples were evaluated as like the reconstituted flakes, in replication two, 30.8% and in replication three, 27.9%. No sample was evaluated as being like the reconstituted flakes in more than 50% of the trials. Only samples 13E, 26-40C, and 13B were judged as similar to the reconstituted flakes in 50% of the trials. Sample 8C1 was the only sample in this series which was never judged as

similar to the reconstituted flakes. The least significant difference between sample means at the 0.05 level was 0.2596. Ranked treatment means for this second preliminary study are shown for each sample in Table 1. There was no significant difference at the 0.05 level between those samples with means ranging from 0.2776 to 0.5000 and those with means from 0.0000 to 0.2223.

The results of the preliminary studies indicated the judges could not detect odor similarity between the oxidized lipid fractions and the "off flavored" sweet potato flakes. Therefore, samples of steam distillate stored under oxygen and under nitrogen were included in the next studies comparing the odors of the oxidized fractions to samples of good and "poor" reconstituted sweet potato flakes. The lipid fractions were not as thoroughly separated in these studies because all similarity to the "off flavor" of the sweet potatoes seemed to have been lost by the thoroughness of chromatographic separation in the preliminary studies.

The revised score card used in the second preliminary study was of little value in the statistical analysis. For this reason the second revised score card was prepared as shown in Appendix A. This score card proved to be easy for the judges to use, and it provided the information needed. This was the score card judges used throughout the remainder of this project.

Similar studies were carried out in July and August and October, 1969 to perfect the methods used. The results of these studies were not identical as can be seen in Tables 2 and 3 in

TABLE 1

RANKED TREATMENT MEANS FOR SECOND PRELIMINARY STUDY

Sample	Means
13E	0.5000
26-40C	0.5000
13B	0.5000
13D	0.4446
8D2	0.4443
8E	0.4443
11B	0.4443
8C3	0.3886
8D4	0.3886
1-15	0.3333
16-23B	0.3333
8C4	0.3333
8C5	0.3333
8F3	0.3333
11C	0.3333
13F	0.3333
26-40B	0.3329
13C	0.3329
26-40E	0.2780
26-40D	0.2776
8D3	0.2776
11A	0.2776
13A	0.2223
41-50	0.2223
16-23A	0.2220
8C2	0.1670
8F1	0.1670
8-B	0.1666
8F2	0.1666
11D	0.1666
26-40A	0.1113
8-A2	0.1113
8-A3	0.1113
8C1	0.0000

Appendix B. Differences in results might be due to variations in the odors of the samples produced and to some changes in personnel on the panels. Despite these variations the overall results were the same, as again none of the samples was evaluated as like the good or "poor" flakes.

The TLC plates prepared from the samples used in the October study showed many of the same compounds were present in all samples. The R_f values calculated from the spot movements on these plates also indicated that complete separation of compounds had not been achieved.

A pure sample of steam distillate proved hard to obtain without some moisture content. Also various important volatile substances were apparently lost during preparation as the final samples did not always have the same odor. The use of the short path molecular still was begun in an attempt to retain more of the sweet potato volatiles than were retained with the use of the higher temperatures of steam distillation.

TLC plates were spotted with samples of distilled and non-distilled cooked and raw sweet potatoes, saponified and non-saponified samples of fraction 8 and the distillate and a beta-ionone standard. This standard was the only pure compound present; all other spots showed that complete separation of pure compounds had not yet been achieved. The standard was used in this series because it had been suggested that beta-ionone was one of the main ingredients in the "off flavor" of sweet potatoes. Apparently there were no beta-ionone-like compounds present in these samples because the standard did not behave like any of the compounds in the isolated fractions.

Oxidized samples of cooked saponified and unsaponified fraction 8 and distillate developed odors which this researcher felt resembled those of the "off flavored" sweet potato flakes. These samples were submitted to a panel of 10 judges for comparison with the odor of reconstituted "poor" sweet potato flakes. As can be seen in Table 4, Appendix B, only the saponified non-distilled fraction 8 and the unsaponified fraction 8 were judged as like the sweet potatoes in over 50% of the trials. Absence of various judges during the test resulted in a great deal of missing data, so no statistical analysis was attempted with this series.

The next set of samples to be evaluated included oxidized samples of raw saponified fraction 8, raw saponified distilled fraction 8, saponified distillate, unsaponified fraction 8, unsaponified distillate, and a sample containing a combination of saponified distilled fraction 8 and saponified distillate. These samples were again compared to samples of reconstituted "poor" sweet potato flakes. A compilation of the judges' scores is shown in Table 5, Appendix B. Only the unsaponified distillate was judged as like the sweet potatoes in over 50% of the trials. No statistical analysis was carried out because there were so few trials in this series.

The epi-phase and hypo-phase of fraction 8 were separated and spotted on TLC plates along with the unseparated fraction 8. The plates again showed that pure compounds had not been obtained. No definite odor developed in either phase during oxidation. Results of judging and chromatographic analysis of fraction 8 indicated that

it was impossible to obtain a pure compound with the methods used. The results also indicated that fraction 8 had been purified to the point where it no longer had an odor resembling that of the reconstituted sweet potato flakes. Therefore, fraction 8 was set aside, and the remainder of this study was centered on the volatiles of the sweet potatoes as removed by short path molecular distillation.

A panel of 15 judges compared the odor of the oxidized distillate, epi- and hypo-phases, good reconstituted sweet potatoes, and a blank to the odor of reconstituted "poor" sweet potato flakes. Statistical analysis of the data obtained showed that the sample of good reconstituted sweet potato flakes was judged as similar to the "poor" reconstituted flakes much more often than was any other sample. The least significant difference between sample means at the 0.05 level in this series was 11.3519. The ranked treatment means and their corresponding Arcsin values are shown in Table 2.

TABLE 2

RANKED TREATMENT MEANS AND ARCSIN VALUES FOR FIRST DISTILLATE SERIES

TRT No.	Treatment	Means	Arcsin
4	Sweet Potato	84.2894	99.1
2	Total Distillate	33.8732	31.1
3	E-Phase	29.2573	23.9
1	H-Phase	28.0736	22.1
5	Blank	12.1266	4.4
LSD at 0.05		11.3519	3.9

Due to the difference in variance at different per cent levels, the means obtained here were converted to the Arcsin constant as given in Appendix Table A16 in Snedecor and Cochran (45). Using these Arcsin values in 99.1% of the trials, the good sweet potato flakes were judged as having an odor similar to that of the "poor" flakes; whereas, the total distillate was so judged in only 31.1% of the trials, the epi-phase in 23.9% of the trials, the hypo-phase in 22.1% of the trials, and the blank in 4.4% of the trials.

The true values of the means plus the least significant difference at the 0.05 level indicated the differences between the distillates and the good sweet potatoes and the distillates and the blank were much greater than could be attributed to chance. However, the differences between the total distillate and the epi-phase, the total distillate and the hypo-phase, and the epi-phase and hypo-phase could be due to chance. By converting to Arcsin values the difference between the total distillate and the epi- and hypo-phases also appears to be significant at the 0.05 level. A summary table of the analysis of variance for this series is given in Table 6, Appendix B.

A blank was included in this series to provide a sample clearly not like the "off flavored" sweet potatoes as a check on the accuracy of the panel. The Hyflo Supercel used in making the blank did have a faint somewhat powdery odor which might have made the judges think there was some odor present. Since the blank was chosen as like the "off flavored" flakes in only 4.4% of the trials, this faint odor apparently did not prove very confusing to the panel.

The graphs from GC analysis of the total distillate and the non-saponifiable fraction of the distillate are shown in Figures 4 and 5.

A comparison of these graphs shows that 13 peaks were removed by saponification. It was concluded that these peaks were esters, as only esters would be removed by saponification. An acid with the solubility characteristic needed to be isolated in this fraction would probably not chromatograph in this manner. All fatty acids with more than 10 carbons need to be esterified for chromatography. Two new peaks appeared after saponification; these were probably alcohols arising from the hydrolyzed esters. The non-saponifiable fraction contained a large number of slow moving compounds which were probably long chain alcohols or possibly polyalcohols. Other compounds present in these samples were various hydro-carbons and cyclic compounds.

The GC analyses showed it was possible to separate the compounds for mass spectral analysis. The GC graph obtained from analysis of the separate distillate sample prepared to send to IFF is shown in Figure 6. This graph and a GC graph containing 4 standards which chromatographed in the same area as the distillate were sent to IFF with the sample. A comparison of Figures 4 and 6 indicates the distillate samples differed mainly in the amount of the fraction placed on the column. The mass spectral analysis indicated that the distillate sample submitted was made up of a complex mixture of previously unidentified compounds which are thought to be mainly complex terpenes.

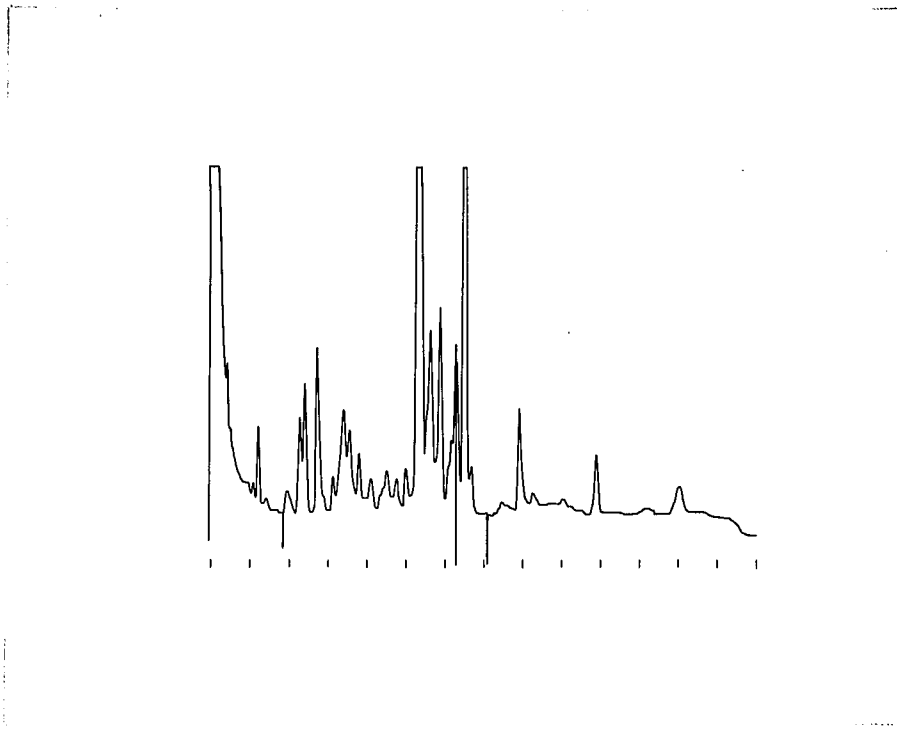

Figure 4

Gas Chromatogram of Total Distillate March 20, 1970

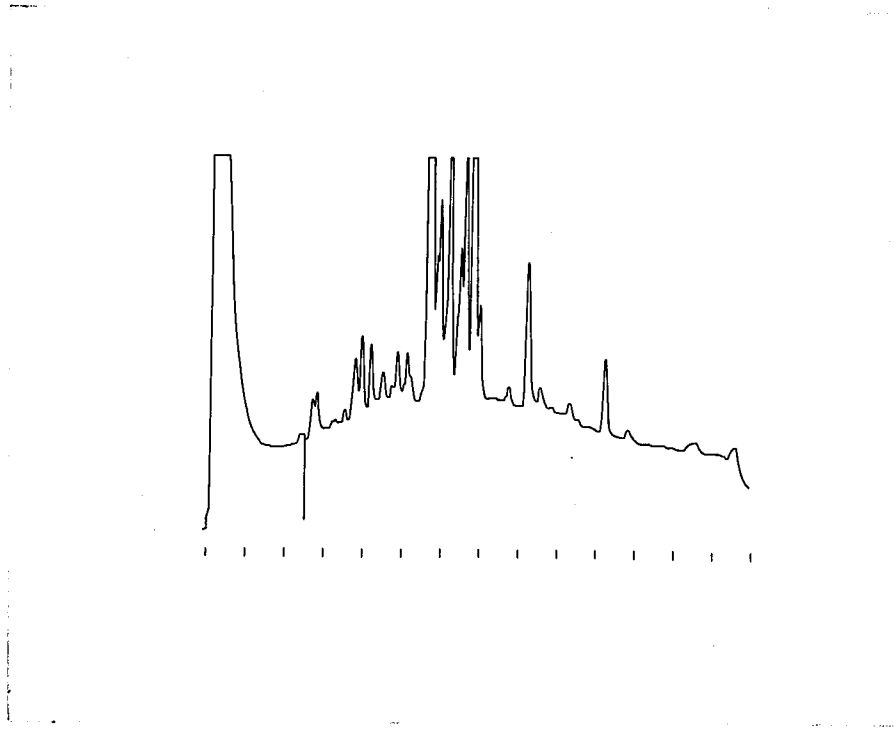

Figure 5

Gas Chromatogram of Non-Saponifiable Distillate March 20, 1970

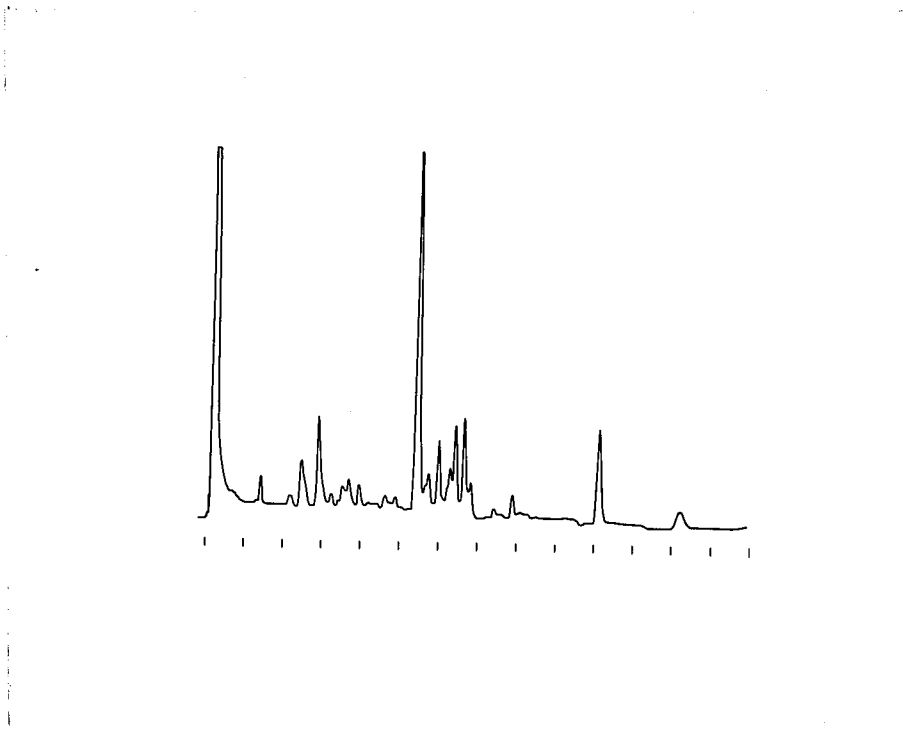

Figure 6

Gas Chromatogram of Distillate Sent to IFF for Analysis April 17, 1970

In an attempt to determine if a mixture of good sweet potato flakes and extracts would produce an odor similar to "off flavored" flakes, good reconstituted sweet potato flakes were added to the extract samples last evaluated by the panel. The panel then compared the odors of these samples plus those of plain reconstituted good flakes to "poor" sweet potato flakes. Statistical analysis of the data obtained showed the least significant difference between sample means at the 0.05 level was 7.1371. The ranked treatment means and their corresponding Arcsin values for this series are shown in Table 3.

TABLE 3

RANKED TREATMENT MEANS AND ARCSIN VALUES
FOR SECOND DISTILLATE SERIES

TRT NO.	Treatment	Means	Arcsin
4	Good Sweet Potatoes	75.0330	93.3
1	Total Distillate+	65.3541	82.6
2	E-Phase+	65.1492	82.3
3	H-Phase+	39.2315	40.0
LSD at 0.05		7.1371	1.6

+ = plus good USDA sweet potatoes

In this series the Arcsin values showed the good sweet potatoes were evaluated as similar to the "poor" flakes in 93.3% of the trials, the total distillate plus good flakes like the "poor" sample in 82.6% of the trials, the epi-phase plus good sweet potatoes like the "poor" sample in 82.3% of the trials and the hypo-phase plus good sweet potatoes like the "poor" sample in 40.0% of the trials. These results indicated the addition of the good sweet potatoes to the oxidized extracts increased their similarity to the "off flavored" flakes. These evaluations clearly showed that some of the same distinct odors present in the "off flavored" sweet potatoes were also present in the good sweet potatoes.

Tests for significant differences in this series would show the same results using the true values or the Arcsin values. Differences between the good sweet potatoes and the extracts with the good sweet potatoes added were all significant at the 0.05 level, indicating

that the good sweet potatoes alone contained more odors like the "off flavored" sample than the good sweet potatoes combined with an oxidized extract. Differences between the selection of the total distillate with good sweet potatoes and the epi-phase with good sweet potatoes were probably due to chance. All other differences between the sample selections in this series were significant at the 0.05 level so they could not be attributed to chance. A summary table of the analysis of variance for this series is given in Table 7, Appendix B.

A sample of the reconstituted commercially prepared sweet potato flakes was added to the previously evaluated series of samples. In an attempt to use flakes with the utmost "off flavor" in these comparisons, "off flavored" flakes which contained no sugar were reconstituted for this series of evaluations. Absence of sugar provides a flake with a very porous structure and great instability thus permitting an accumulation of extreme "off flavor." Statistical analysis of the data obtained showed the least significant difference between sample means in this series at the 0.05 level was 16.3775. The ranked treatment means and their corresponding Arcsin values are shown in Table 4.

The Arcsin values show the USDA processed sweet potatoes were evaluated as similar to the "poor" flakes in 75.5% of the trials. The commercially processed good sweet potatoes were so evaluated in 66.7% of the trials. The total distillate plus good flakes was evaluated as like the "poor" sample in 64.0% of the trials, the hypo-phase plus good flakes like the "poor" sample in 62.8% of the

trials,and the epi-phase plus good flakes like the "poor" sample in 54.2% of the trials. Again the greatest similarity of the tested samples to the "poor" sample appeared in the good sweet potato flakes processed by the USDA. The commercially processed good sweet potato flakes were chosen as similar to the "poor" sample more often than were any of the samples containing extracts plus good sweet potato flakes.

TABLE 4

RANKED TREATMENT MEANS AND ARCSIN VALUES
FOR THIRD DISTILLATE SERIES

TRT No.	Treatment	Means	Arcsin
5	Good Potato, USDA	60.3070	75.5
2	Good Potato, Comm.	54.7544	66.7
3	Total Distillate+	53.0984	64.0
1	H-Phase+	52.4129	62.8
4	E-Phase+	47.4129	54.2
LSD at 0.05		16.3775	8.0

+ = plus good USDA sweet potatoes

If the true value of the means were used for comparisons in this series, there would be no significant differences between any of the sample means at the 0.05 level. With Arcsin values a significant difference at the 0.05 level is obtained between the selection of the USDA processed good sweet potatoes and all other samples. A significant difference is also found between the selection of the commercially prepared sweet potatoes and the epi-phase plus good sweet potatoes. No other differences in this series were significant

at the 0.05 level. A summary table of the analysis of variance for this series is given in Table 8, Appendix B.

Results of the sensory evaluations at this point indicated that the samples most like the reconstituted "poor" sweet potato flakes were the reconstituted good sweet potato flakes. Odors that this researcher could not relate to the "off flavored" sweet potatoes were subsequently so related by panel evaluation. Odors that were very offensive to this researcher did not appear to be offensive to panel members. Many of the judges seemed quite confused when they attempted to relate the odor of the chemical extracts to the odor of the reconstituted sweet potatoes; they seemed much less confused when they tried to evaluate the similarity of the odor of good flakes to that of the "poor" flakes.

This investigator then concluded that it would be of value to try to determine if an untrained panel could distinguish between seasoned samples of reconstituted good and "poor" sweet potato flakes. The samples were presented as a triangle test and in 26 of the 27 trials the judges indicated they felt there was a difference in the samples presented. However, these judges showed a great deal of confusion when they attempted to determine which sample was the different one. The students who comprised this panel were not familiar with sweet potatoes in general except in the baked or candied form; nevertheless most of them expressed an overall liking for the "new" product. The students considered the texture of the reconstituted flakes as mediocre and frequently described it as

sticky or pastelike. The flavor of the products was more objectionable to the students than was the texture. Despite the objections, discussions after the evaluation sessions revealed that the students did not feel a strong dislike for the "off flavored" samples. The opinion was expressed that without a comparison the seasoned "off flavored" product might have been judged as acceptable in a high percentage of the trials.

CHAPTER V

SUMMARY AND CONCLUSIONS

In accordance with the recommendations of Jones (20) and Cox (21) the carotene fractions implicated in the development of "off flavor" in dehydrated sweet potato flakes were thoroughly studied. These fractions were further separated by column chromatography with different absorbents and by preparative and thin layer chromatography. Partition fractions from sweet potato lipids were obtained and subjected to the same treatment as the carotene fractions. No pure compounds were obtained. All fractions were found to contain vast amounts of non-carotene substances with many different compounds which smeared or blurred on preparative chromatography plates.

After oxidation, samples were compared to reconstituted sweet potato flakes by a sensory panel following the procedures developed by Jones (20) and Cox (21) with appropriate modifications. Panel evaluations did not indicate that any of the purified samples had odors similar to those of reconstituted sweet potato flakes. The fractions had apparently been so purified that all resemblance to "off flavor" had been lost.

A steam distillate was next prepared from reconstituted sweet potato flakes. Chromatographic analysis again revealed that no pure compounds had been obtained. Sensory evaluation of oxidized steam

distillate samples showed little correlation between the odor of the samples and the odor of the reconstituted flakes.

Infrared spectrosopy of the steam distillate and fraction 8 failed to verify the presence of C=C, and indicated that carbonyls were minor constituents of these fractions.

Distillate samples obtained with a short path molecular still were subjected to chromatographic analysis before oxidation and presentation to a panel for sensory evaluation. GC analysis of these samples showed that the compounds could be separated for mass spectral analysis. The mass spectral analysis revealed that the distillate sample contained a complex mixture of previously unidentified compounds.

Sensory evaluations of purified fractions continued to show little correlation between sample odor and the odor of reconstituted "poor" sweet potato flakes until reconstituted good sweet potato flakes were added to the purified fractions. Sensory evaluations then revealed some definite similarities of odor between the combined samples and the "poor" sweet potato flakes.

The final sensory evaluation with an untrained taste panel revealed difficulty in selecting the different sample in a triangle test involving seasoned reconstituted good and "poor" sweet potato flakes. The panel members also indicated that the "poor" sample was not highly objectionable to them.

The results obtained throughout this study led to the following conclusions:

1. The lipid and volatile fractions from sweet potatoes are highly complex compounds which cannot be separated into pure substances with present analytical methods.

2. Purification of compounds implicated in "off flavor" development results in a loss of any resemblance to "off flavored" sweet potato flakes.

3. Development of "off flavor" in sweet potato flakes is a highly complex reaction involving combinations of as yet unidentified compounds.

4. Many of the same odors present in "off flavored" sweet potato flakes are also present in good sweet potato flakes.

5. "Off flavored" sweet potato flakes are not as objectionable to the layman as they are to professionals studying the product.

6. Oxidation of sweet potato fractions may reach a point beyond which all resemblance to "off flavored" sweet potato flakes is lost.

CHAPTER VI

RECOMMENDATIONS FOR FURTHER INVESTIGATION

The results obtained in this project indicate continued attempts should be made to identify the compounds in sweet potato distillate revealed by GC and mass spectral analysis. Continued attempts should also be made to identify the compounds in lipid fraction 8 by use of mass spectral analysis.

As the "off flavor" development does not seem to be associated with any one compound, another investigation should involve the combination of various fractions in an attempt to determine if any of the combinations develop the characteristic odors of "off flavored" sweet potato flakes.

The "poor" sweet potato flakes used in this study had been stored in air by the USDA so that "off flavors" would develop. These products should be much more "off flavored" than commercially processed sweet potato flakes obtained from grocers' shelves. A study to determine the amount of "off flavor" developed in commercial products after storage in comparison to the USDA product stored in air should give some indication of consumer acceptance.

The sweet potato varieties used in this study were the Goldrush and Centennial which have similar characteristics. Other varieties of sweet potatoes should also be studied to determine

whether they lend themselves as well to the making of flakes and if they are acceptable to consumers when reconstituted. Variations in flake samples from different processors and between samples from the same processor should also be investigated.

Because none of the purified fractions was evaluated as like the reconstituted sweet potato flakes and the panels did not seem to object strongly to any of the oxidized fractions, a study of the complete product seems warranted. Samples of good and "poor" USDA processed sweet potato flakes should be evaluated in comparison with commercially processed flakes. Samples should be submitted to a large consumer panel, as well as the small trained panel. The products should be used in the pure reconstituted form, as well as in seasoned samples and in recipes such as those for sweet potato casseroles, soufflés, biscuits, and pies to determine if added ingredients mask the "off flavor" development and increase consumer acceptability.

Sweet potato products should also be developed as a type of nutritious snack food, especially for use of school age children.

The oxidation of sweet potato flakes is a progressive reaction. Studies should be carried out to determine at what point in storage the commercially prepared product becomes so oxidized that it ceases to exhibit the odors associated with "off flavored" sweet potatoes.

The sweet potato industry needs to attempt to educate more consumers to use sweet potatoes; to change the image of the product from that of a poor man's food to that of an interesting and nutritious product of value to all economic groups. Consumer acceptability

studies could be conducted in various parts of the country especially in areas where consumers may not be familiar with the use of sweet potatoes. Studies should also be conducted to determine if consumers can recognize the difference between varieties of sweet potatoes.

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APPENDIX A

SCORE CARDS, JUDGE'S INSTRUCTIONS AND PERSONAL DATA SHEET

DATA SHEET USED FOR JUDGES IN

PRELIMINARY EVALUATIONS

PERSONAL DATA

Name:_____

Address:_____

Phone Number:_____

Age:_____

Occupation:_____

Department or Major:_____

Experience in Test Panels:_____

_____

Health: Good_____ Fair_____ Poor_____

Do you smoke? Yes_____ No_____

Do you like sweet potatoes?

Very well_____ Moderately well_____ Fairly well_____

Any sessions you know of you will have to miss? (List dates)

INSTRUCTIONS FOR JUDGES AT PRELIMINARY EVALUATION ONE SESSIONS

1. Smell the samples--rely on first sensations in evaluating each sample.

2. Pause a few seconds before smelling each sample.

3. Score cards are present on each tray for each test. Pick up one. Write your name and the date on it; complete it and turn the score card face down. If you have any comments about the samples, please add them at the bottom or on the back of the score card.

4. If you smoke, please refrain from smoking shortly before your evaluation sessions.

5. Please do not talk or make any unnecessary facial expressions during the evaluation sessions.

6. If you become fatigued, wait a minute or two before continuing. You will recognize fatigue as the sensation when all samples smell alike.

SCORE CARD FOR PRELIMINARY EVALUATION ONE

Name_____

Date_____

PAIRED TEST FOR ODOR DETECTION

Circle one word below which describes the relationship of the odor of the two samples.

ALIKE DIFFERENT

INSTRUCTIONS AND SCORE CARD FOR PRELIMINARY EVALUATION TWO

JUDGING SESSION--INSTRUCTIONS

You will be presented with two sets of samples, one tray at a time. Sample X is reconstituted sweet potato flakes. The balance of the coded samples are to be compared in odor to Sample X, and scored as being similar or dissimilar to the sweet potatoes, or as having no odor at all.

Indicate on the score card 1-3 for degree of similarity to sweet potatoes, 4 if it is not really similar but may be a component of the total odor, 5 if no odor is present, or 6-8 for degree of dissimilarity from the sweet potatoes.

Allow approximately 30 seconds between evaluating one set of samples and the next set. Please do not talk during the testing, as unnecessary noise may influence the response of others. Please recap the vials after sniffing.

SCORE CARD

Test_____

Name_____

Date_____

Scale	Comment	Coded Numbers		
1	Extremely like Sample X			
2	Moderately like Sample X			
3	Slightly like Sample X			
4	Not really similar but may be a component of the total odor			
5	No odor			
6	Slightly different from Sample X			
7	Moderately different from Sample X			
8	Extremely different from Sample X			

JUDGE'S SCORE CARD USED THROUGHOUT ACTUAL STUDY

Name_____

Date_____

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

Alike	Different
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

SCORE CARD USED IN TRIANGLE TESTS OF SEASONED

RECONSTITUTED SWEET POTATO FLAKES

Name_____ Date_____

Is there a difference in the samples presented?_____

If so, which one is different?_____

Which sample (samples) do you prefer?_____

Describe the texture of the product - check at least one word in each column.

good_____	slimy_____
mediocre_____	grainy_____
poor_____	runny_____
	sticky_____
	paste-like_____
	_____

APPENDIX B

COMPILATION OF JUDGES' SCORES

ANALYSIS OF VARIANCE

TABLE 1

COMPILATION OF JUDGES' SCORES FOR
PRELIMINARY EVALUATION ONE

Fraction	Comparison		Compiled Judgings: Alike	Different	No Odor	Partition Fraction	Comparison		Compiled Judgings: Alike	Different	No Odor
2B	1	G	6	7		1-5	1	G	2	14	
2F	2	G	4	11		41-50	2	G	2	13	
5C	3	G	2	8	4	11-15	3	G	7	7	2
6B	4	G	4	9	1	21-25	4	G	6	9	
6C	5	G		15		31-35	5	G	3	12	
7A	6	G	2	10	2	6-10	6	G	2	14	
7B	7	G	2	10	2	16-20	7	G	8	6	1
7C	8	G	3	11		26-30	8	G	5	10	
7D	9	G	2	13		36-40	9	G	2	13	
7E	10	G	4	9	1	1-5	10	P	6	9	
2B	11	P	6	7	1	41-50	11	P	1	14	
2F	12	P	5	10		11-15	12	P	3	9	3
5C	13	P	2	9	3	21-25	13	P	5	10	
6B	14	P	5	7	3	31-35	14	P	8	8	
6C	15	P	1	14		6-10	15	P	4	11	
7A	16	P	3	7	4	16-20	16	P	9	6	
7B	17	P	2	10	2	26-30	17	P	8	7	
7C	18	P	2	10	2	36-40	18	P	2	13	
7D	19	P	3	11							
7E	20	P	2	9	3						

TABLE 2

COMPILATION OF JUDGES' SCORES FOR
JULY, AUGUST, 1969 PANEL

Name Good Sweet Potato Flakes

Date July 30, August 1, 4, 1969

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike		Different	
	Code	Score	Code	Score
Steam dist. - N_2	SDN	8	SDN	35
Steam dist. - O_2	SDO	9	SDO	34
Fraction 8	8	6	8	37
Fractions 9-12	9-12	7	9-12	36
Fraction 13	13	21	13	22

Name "Poor" Sweet Potato Flakes

Date July 30, August 1, 4, 1969

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike		Different	
	Code	Score	Code	Score
Steam dist. - N_2	SDN	7	SDN	36
Steam dist. - O_2	SDO	4	SDO	39
Fraction 8	8	9	8	34
Fractions 9-12	9-12	7	9-12	36
Fraction 13	13	16	13	27

TABLE 3

COMPILATION OF JUDGES' SCORES FOR
OCTOBER, 1969 PANEL

Name Good Sweet Potato Flakes

Date October, 1969

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike		Different	
	Code	Score	Code	Score
Steam dist. - O_2	549	18	549	30
Fraction 8	608	20	608	28
Steam dist. - N_2	689	23	689	25
Fraction 13	752	7	752	41
Fractions 9-12	803	16	803	32

Name "Poor" Sweet Potato Flakes

Date October, 1969

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike		Different	
	Code	Score	Code	Score
Steam dist. - O_2	549	22	549	26
Fraction 8	608	20	608	28
Steam dist. - N_2	689	22	689	26
Fraction 13	752	8	752	40
Fractions 9-12	803	14	803	34

TABLE 4

COMPILATION OF JUDGES' SCORES FOR
JANUARY, 1970 PANEL

Name Saponified

Date Composite - January, 1970

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples "alike" - those with no similarity as "different".

	Alike Code	Alike Score	Different Code	Different Score
Sap. un-chrom. dist.	415	2	415	23
Sap. 8	635	12	635	13
Sap. chrom. dist.	850	4	850	21
Sap. non-dist.	970	16	970	9

Name Non-saponified

Date Composite - January, 1970

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike Code	Alike Score	Different Code	Different Score
Un-sap., un-chrom. dist.	205	3	205	23
Un-sap. 8	319	16	319	10
Un-sap. chrom. dist.	511	2	511	24
Un-sap. non-dist.	742	13	742	13

TABLE 5

COMPILATION OF JUDGES' SCORES FOR
FEBRUARY, 1970 PANEL

Name Saponified

Date February, 1970

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike Code	Alike Score	Different Code	Different Score
Saponified 8	104	10	104	16
Sap., dist. 8	425	3	425	23
Sap., distillate	679	8	679	18

Name Non-saponified & Combination

Date February, 1970

Test for Odor Detection

Compare the samples in the vials to samples X or Y. List those that are in any way similar to the samples as "alike" - those with no similarity as "different".

	Alike Code	Alike Score	Different Code	Different Score
Un-sap. 8	255	8	255	11
Un-sap. dist.	390	13	390	6
Sap. dist. 8 + Sap. dist.	760	3	760	16

TABLE 6

ANALYSIS OF VARIANCE FOR FIRST DISTILLATE SERIES

Source	df	SS	MS
Total	14	9376.6302	
Replications	2	76.7883	38.3941
Treatment	4	9009.0338	2252.2584*
Expt. Error	8	290.8079	36.3509

*Sig. at 0.01 level

TABLE 7

ANALYSIS OF VARIANCE FOR SECOND DISTILLATE SERIES

Source	df	SS	MS
Total	11	2200.4354	
Replications	2	3.4172	1.7086
Treatment	3	2120.4546	706.8182*
Expt. Error	6	76.5635	12.7605

*Sig. at 0.01 level

TABLE 8

ANALYSIS OF VARIANCE FOR THIRD DISTILLATE SERIES

Source	df	SS	MS
Total	14	1175.2088	
Replications	2	311.1507	155.5753
Treatment	4	258.7710	64.6927
Expt. Error	8	605.2870	75.6608